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Chemical fluctuation and phase transition in amorphous micro silica

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Amorphous micro silica (MS) has been widely applied in various industries, e.g., in cement and calcium silicate production. Porous calcium silicate is an excellent insulation material, especially in high temperature environment. Besides lime, MS is the second key component for producing porous calcium silicate. Recently we have found that the quality, characteristics and source of MS has a strong impact on the performances of the final products. To understand the mechanism of such impact and to improve the properties of final product, it is crucial to identify the difference in morphology, phase transition, impurity among MS materials that originate from different sources and/or are produced using different methods. In this study, we investigate three types of MS materials with different impurities, which were obtained from three producers. Two of the MS are by-products from the silicon production (MS1 and MS2) and the third is a by-product from the ferro silicon production (MS3). Their chemical compositions (wt%) were analysed by means of X-ray fluorescence (XRF) (see Table 1).

Table 1. Chemical compositions (wt%) of amorphous micro silica (MS) from three production sources

Sample	SiO_2	MgO	Na_2O	Fe_2O_3	T_{exo} (°C)	T_{endo} (°C)
MS1	95.9	0.3	0.04	0.08	1107	226
MS2	96.1	0.2	-	0.07	1184	248
MS3	92.1	1.2	0.50	0.70	1104	238

Through differential scanning calorimetry (DSC) we have identified striking differences of phase transition behavior among the three samples. During the first upscan, the exothermic response peak of MS2 occurs at a much higher temperature (T_{exo}) compared to MS1 and MS3 and °C (Fig. 1 and Table 1). These peaks are attributed to the cristobalite formation. This can be confirmed by the second upscan curves showing the endothermic peaks in the temperature region of T_{endo}=226~248˚C (Inset of Fig. 1 and Table 1), i.e., the temperature region of the α- to β-cristobalite transition. The crystal structure of MS is identified using X-ray diffraction (XRD) on both the as-received (Fig. 2), and the heat-treated samples at their respective T_ps for 3 hours (Inset of Fig. 2).

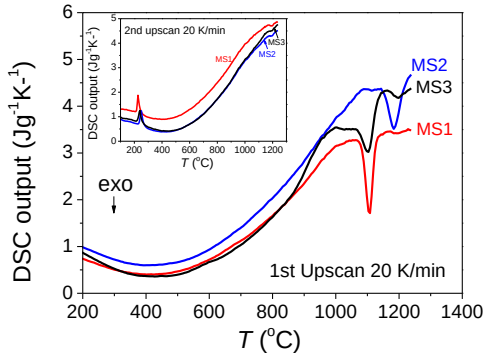

Figure 1: DSC output of MS1, MS2 and MS3 for the 1st upscan. Inset: for the 2nd upscan.

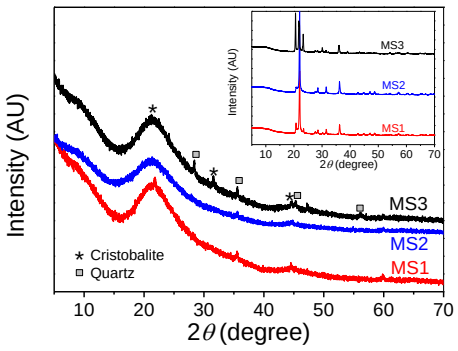

Figure 2: XRD spectra of both the as-received and the heat-treated samples. Intensity: arbitrary unit (AU)

The as-received silica contains mainly amorphous phase with a trace of quartz. MS3 has a slightly higher degree of crystallinity than MS1 and MS2, indicating that MS3 has a higher content of impurities as nucleating agents. MS1 exhibits higher content of cristobalite than MS2. All the heat-treated samples display cristobalite, but MS3 also exhibits traces of other crystalline phases.

The higher T_{exo} of MS2 indicates the presence of fewer crystallites in MS2 compared to MS1 and MS3. The XRD spectra indicate that a trace of cristobalite is already present in the as-received MS1 and MS3 samples, and acts as nucleating agent resulting in lower T_{exo}. MS3 has a higher content of impurity, especially higher Na and Fe contents in than MS1 and MS2. The devitrification of amorphous micro silica is strongly enhanced by its impurities, especially alkali metals. Micro silica originated from different production sources exhibits different morphology and structure, and hence could affect the properties of calcium silicate products. This is being closely investigated in a separated work. This work indicates that the detection of crystallization from DSC scans could be used as a standard method to monitor the fluctuation of the impurities of amorphous micro silica.